Final Report - Evaluation and Compilation of Neutron Activation Cross Sections for Medical Isotope Production

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1. Project Summary

Goal–This NEER project involved the calculational assessment and experimental verification of certain neutron cross sections that are related to widely needed new medical isotopes. Experiments were performed at the Oregon State University (OSU) TRIGA Reactor (OSTR) and the High Flux Irradiation Reactor (HFIR) at Oak Ridge National Laboratory (ORNL).

Specifically the cross sections associated with the production of ^{103}Pd, ^{195m}Pt, ^{117m}Sn, ^{67}Cu, ^{188}W/^{186}Re, ^{166}Ho, ^{47}Sc, ^{91}Y, and ^{225}Ac/^{213}Bi were investigated.

This project was conducted in one continuous phase with eight tasks, some of which overlapped in time. As the project developed, tasks 5 and 6 were merged, since they involved similar measurements at the two different locations, OSU and ORNL. Summaries of the tasks and associated results are given below. The accomplishments are listed separately in Section 3 by medical isotope, since each of the tasks was performed similarly for each isotope investigated and this manner of reporting allows a clearer picture of what was accomplished.

This report constitutes the final project report for the entire project. A Ph.D. dissertation covering the ORNL portion of this work is currently in draft form and will be issued soon. Undergraduate theses written by several of the students participating in the project and earlier publications associated with this project are listed in Section 5 below. Other publications are under consideration as the results are further analyzed, pending determination that the results meet acceptability criteria for inclusion in standard cross section libraries.

2. Summary of Objectives

Task 1: Identify isotopes to be evaluated

In addition to the cross sections related to the five medical isotopes listed in the proposal, previous studies will be used to determine other badly needed cross section measurements in support of the production of projected high demand medical radioisotopes at a reasonable cost. Lack of data or available integral data for these isotopes will be assessed to determine which of these isotopes have sufficient data for cross section determinations. It is anticipated that up to five cross sections associated with other medical isotopes can be investigated beyond the five initially chosen.

Task 2: Perform scoping calculations and prepare targets

Calculations using MCNP (and other computer codes, as necessary) will be performed to determine the required target compositions and configurations and the necessary target irradiation times for production of the desired medical isotopes. Target material will be procured and final design and encapsulation of the targets performed.

Task 3: Irradiate targets in different irradiation facilities in the OSTR

The optimum positioning of the targets with respect to irradiation locations with differing neutron spectra for activation will be determined. Samples will be irradiated in up to four OSTR irradiation facilities with differing neutron spectra.

Task 4: Use gamma spectrometry to determine activities

Gamma spectrometry will be performed on the irradiated targets using OSU Radiation Center equipment to determine induced activity, which will in turn be used to derive cross section values.

Task 5: Determine neutron cross sections from activity measurements

Data generated in Task 4 will be used to determine the thermal and resonance integral cross sections or the (n,p) cross sections of the key radionuclides with missing or inferior cross section values. This activity will include a thorough error propagation analysis and statistical analysis for each measured cross section.

Task 6: Unfold prior integral cross section data

Cross section data at ORNL will be unfolded to determine thermal cross sections and resonance integrals for the isotopes related to the medical isotopes of interest identified in this study.

Task 7: Compare experimental results with calculated cross sections

The cross sections calculated from the OSTR experiments will be compared to those determined from the ORNL integral data. From these authenticated cross sections, production rates for the associated medical isotopes will be calculated for various U.S. production sites.

Task 8: Document results and submit for publication

Documentation must be adequate to meet acceptability criteria for inclusion in standard cross section libraries. A full report of the entire project will be prepared to satisfy this requirement and to serve as the basis for a Ph.D. dissertation. Results will also be submitted for publication in appropriate journals

3. Accomplishments

3.0 General considerations

Previous studies were used to determine badly needed cross section measurements in support of the production of projected high demand medical radioisotopes at a reasonable cost. Lack of data or available integral data for these isotopes was assessed to determine which of these isotopes have sufficient data for cross section determinations.

Project team members discussed which of the poorly known or unknown medical isotope cross sections were most likely to be successfully measured. Final determination of isotopes from among the candidates was influenced by the availability of existing data, the cost of target materials, and the likelihood of achieving adequate production levels of the isotopes. The final list of medical isotopes included ^{103}Pd, ^{195m}Pt, ^{117m}Sn, ^{67}Cu, ^{188}W/^{186}Re, ^{166}Ho, and ^{47}Sc measurements on the OSTR and ^{188}W/^{188}Re and ^{225}Ac/^{213}Bi on the HFIR. The HFIR measurements were conducted under the subcontract to the University of Maryland in conjunction with ORNL personnel. Upon further reflection and calculation, a couple of the originally proposed medical isotopes were excluded from this study. Specifically, ^{125}I was not considered because of the large potential for radioactive contamination working with radioactive ^{125}Xe gas and volatile ^{125}I.

Calculations were performed to determine the required target compositions and configurations and the necessary target irradiation times for production of the desired medical isotopes. MCNP was used to calculate reaction rates for input to the nuclide equations. These calculations took into consideration the neutron spectrum of the OSTR and HFIR. The nuclide equations, which in some cases were long or multi-branched, were solved for all the radionuclides involved, in order to make the proper time corrections for irradiation, decay, and counting interval.

A general solution for all possible source and loss terms was developed with analytical solutions for an arbitrary target material and subsequent progeny [Binney]. A literature search is being conducted to see if such a paper has been previously published. A spreadsheet was developed to solve the nuclide kinetic equations for the chains of interest.

The MCNP [Breismeister] code was used to investigate self-shielding effects in the target foils. These calculations were compared to analytical calculations.

Target material was procured by purchase of high purity foils and a specially designed set of tungsten concentric annular cylinders. Final design and encapsulation of the targets was completed. Target acceptability for irradiation in the OSTR and HFIR was verified by performing reactivity calculations, evaluating temperature limits, and determining target compliance with Technical Specifications for experiments.

The optimum positioning of the targets with respect to irradiation locations with differing neutron spectra for activation was determined. Samples were irradiated in three different OSTR irradiation facilities with differing neutron spectra, namely, the thermal column (thermal neutron spectrum with less than 1% non-thermal neutron contribution), the Cadmium-Lined In-Core Irradiation Tube (CLICIT), which has less than a 1% thermal neutron component, and the In-Core Irradiation Tube (ICIT), which has a full neutron spectrum present. The ICIT and CLICIT, which are specifically designed for application of the cadmium difference method, were used to calculate thermal and epithermal fluxes. At the HFIR samples were irradiated in the Hydraulic Tube (HT) and Peripheral Target Position (PTP). Different neutron spectra were used for activation since both thermal neutron activation and burnup cross sections and also resonance integrals were desired to be measured.

Gamma spectrometry was performed on all irradiated targets, including flux monitors, to determine induced activity, which in turn allowed the calculation of cross section values. At OSU samples were counted on a hyperpure germanium semiconductor detector (27% efficient relative to NaI, 1.87 keV resolution at 1322 keV) coupled to a digital, high stability multichannel analyzer. At ORNL samples were counted on an18.8% efficient, 1.80 keV hyperpure germanium semiconductor detector.

At ORNL after irradiation, the capsules were removed, transported to a hot cell, and cut open. The quartz ampoules containing monitor samples were removed and transported to a laboratory where the ampoules were soaked in concentrated HNO_3 for a few minutes, then rinsed with water to remove surface contamination, dried and mounted on counting cards. The gamma spectra of the irradiated samples were measured with no chemical processing.

Single and double radiative capture reactions were used to determine the axial flux profile at one of the six PTPs. The results for the various reactions in each capsule were averaged to produce a single value for thermal flux for each array position. The flux monitors involving a single radiative capture reaction included: $^{113}In(n,()^{114m}In$, $^{109}Ag(n,()^{110m}Ag$, $^{58}Fe(n,()^{59}Fe$, $^{116}Sn(n,()^{117m}Sn$, $^{112}Sn(n,()^{113}Sn$, and $^{122}Te(n,()^{123}Te$. The measured thermal neutron flux values ranged from $1.11x10^{15}$ to $1.49x10^{15}$ n cm^{-2} s^{-1}, and the epithermal neutron flux values ranged from $3.5x10^{13}$ to $7.5x10^{13}$ n cm^{-2} s^{-1}.

In the PTP a neutron inelastic reaction, $^{117}Sn[n,n']^{117m}Sn$, was used to determine the integrated neutron flux above the calculated threshold (0.318 MeV) for this reaction. The measured flux values were $5.87x10^{14}$ and $5.99x10^{14}$ n cm^{-2} s^{-1} for positions 4 and 5 of the PTP, respectively. These flux values were based on a 222 ± 16 mb cross section for the $^{117}Sn[n,n']^{117m}Sn$ reaction

which was previously measured in the HFIR hydraulic tube [Mirzadeh et al. 1997], and a value of 0.60 for the ratio of fast (E_n = 0.318 MeV) to thermal flux obtained from PTP design data.

Data generated in Task 4 was used to determine the thermal and resonance integral cross sections or the (n,p) cross sections involved in the production of the medical isotopes mentioned previously. Flux monitor foils with known resonance integrals were used to calculate the epithermal flux. Then flux monitor foils with known thermal absorption cross sections were used to calculate the thermal flux.

Two types of flux monitors were used in the OSTR. $^{58}Fe(n,()^{59}Fe$ was used to unfold the thermal and epithermal flux using the cadmium difference method. $^{54}Fe(n,p)^{54}Mn$, $^{58}Ni(n,p)^{58}Co$, and $^{60}Ni(n,p)^{60}Co$ reactions were used to unfold the fission spectrum. Measured values were $9.95x10^{11} \pm 1.34x10^{11}$ n cm^{-2} s^{-1} for the epithermal flux in the CLICIT, compared to the historical value of $1.0x10^{12}$ n cm^{-2} s^{-1}. Thermal neutron flux values measured in the ICIT ranged from $9.11x10^{12}$ to $1.22x10^{13}$ n cm^{-2} s^{-1}, compared to the historical value of $1.0x10^{13}$ n cm^{-2} s^{-1}.

3.1 ^{103}Pd measurements

The cross section for neutron capture by ^{102}Pd to form ^{103}Pd ($t_{1/2}$ = 17 d) was measured using samples of naturally occurring Pd metal, in which the abundance of ^{102}Pd is only 1.0%. The presence of ^{103}Pd is signaled by the emission of two gamma rays, 357 keV (0.04% branch) and 497 keV (0.002% branch). Despite the small gamma ray branching ratios and low abundance of ^{102}Pd, the presence of ^{103}Pd in irradiated samples was easily observed. However, the measured ratio (~3:1) of the intensities of the 357 keV and 497 keV gammas differed from the 5:1 value expected based on the known parameters of the ^{103}Pd decay, suggesting the presence of an impurity in the source. The 357 keV gamma-ray decayed with the expected 17-day half life, but the 497 keV gamma had a much longer decay, in the range of 30-40 days. A careful analysis of the 497 keV line in the gamma ray spectrum showed a width that was no larger than that observed for other gammas in that region, suggesting that the energy of the impurity gamma ray was virtually identical to that of the ^{103}Pd 497 keV transition. It is believed that this impurity is due to a small amount of ^{103}Ru in the sample, produced by neutron capture in ^{102}Ru which must present as a chemical impurity in the Pd metal. Both ^{103}Ru and ^{103}Pd decay to ^{103}Rh through the identical 497 keV gamma ray. Fortunately, the emission of the 357 keV gamma ray is negligible in the ^{103}Ru decay. Thus, while it would be preferable to use two or more gammas in the spectrum to confirm the analysis of the amount of ^{103}Pd activity, only the 357 keV line was used.

Irradiations were carried out in the OSTR thermal column and ICIT. The thermal column irradiation yielded a cross section of 1.3 $\pm$ 0.1 b, while the ICIT irradiation yielded 4.3 $\pm$ 0.4 b. This discrepancy suggests perhaps a significant influence of the resonance region on the ICIT measurement. These measurements are in the process of being repeated to verify these results. Also an irradiation in the CLICIT will be made to try to measure the resonance integral directly.

The accepted literature value of this cross section is 4.8 barns, based on a 1953 measurement using ^{108}Pd as a reference. The subsequent change in the reference value in the ensuing years forces a recalculation of the ^{102}Pd cross section to about 3 barns, which still differs significantly

from the measured thermal value in these experiments. It is possible that this earlier measurement also suffers from the influence of resonance capture.

These measurements served as the basis of the senior thesis of Christopher Duncan, an OSU physics major who completed his degree in June 2003.

3.2 ^{195m}Pt measurements

Natural platinum targets were used to measure the probabilities to form the isomeric states of ^{195m}Pt (4.0 d) by radiative neutron capture and by inelastic neutron scattering. To try to distinguish between the formation of these activities by radiative capture and inelastic scattering irradiations were performed in the thermal column, the ICIT, and the CLICIT. The irradiations produce a multitude of activities in addition to the desired ones. Analysis of these results is in progress.

In part, this work formed the basis of the senior thesis of OSU physics major Rachel Bartlett, who completed her degree in June 2002.

3.3 ^{117m}Sn measurements

Similar to the ^{195m}Pt study, natural tin targets were used to measure the probabilities to form the isomeric states of ^{117}Sn^{m} (13.6 d) by radiative neutron capture and by inelastic neutron scattering. Analysis of these results is also in progress.

This work formed the other part of the senior thesis of OSU physics major Rachel Bartlett, who completed her degree in June 2002.

These measurements were also analyzed by Joseph Coleman as part of his M.S. thesis in Radiation Health Physics. The small capture cross section of ^{116}Sn to the metastable state, ^{117m}Sn, did not present a problem as adequate time (2 days) was allowed for decay to the ground state. Using the cadmium difference method permitted isolation of both a resonance integral and an average thermal neutron cross section.

The epithermal resonance integral measurement resulted in a value of 1.4 ± 0.23 b (compared to the ENDF/B-VI value of 0.49 ± 0.16 b). The 159 keV line of ^{117m}Sn was adjacent to the 156 keV ^{59}Fe line which made resolution somewhat difficult. It was decided that both could not be resolved so the more prevalent of the two was analyzed. More analysis is necessary to further resolve this discrepancy.

3.4 ^{67}Cu measurements

The ^{67}Zn(n,p)^{67}Cu reaction is an exothermic threshold reaction (Q = 0.2058 MeV). However, production of ^{67}Cu in a thermal reactor is difficult due to its very low (~1 mb) thermal neutron cross section.

An enriched (94.60% ^{67}Zn) zinc sample was irradiated at full power (1 MW) for three hours in the CLICIT and ICIT.

After a long decay time (~ 9 days) the ICIT and CLICIT fission average cross sections were measured to be 1.06 ± 0.12 mb and 1.07 ± 0.11 mb, respectively. Mirzadeh et al. (1986) measured the fission average cross section to be 1.23 mb, and noted that this was in "fair agreement with the adopted value of 1.07 mb." O'Brien (1969) experimentally determined the ^{67}Zn(n,p) fission average cross section to be 0.82 ± 0.04 mb.

3.5 ^{188}W/^{188}Re measurements

These measurements were conducted at OSU and more extensively at ORNL. The ORNL results are discussed here.

Neutron self-shielding in the production of ^{188}W via neutron irradiation of ^{186}W by double capture was studied theoretically and experimentally. Tungsten-188 is the parent isotope of ^{188}Re which is being used in dozens of research protocols around the world for a wide range of applications in nuclear medicine, including therapy for cancer, coronary artery disease, and arthritis, and also bone pain palliation. Production in the HFIR at ORNL has typically resulted in ^{188}W yields that are about half of what would be expected based on theoretical calculations using published neutron data. Experiments were performed to validate the neutron cross sections involved in this double neutron capture production scheme and to evaluate neutron self-shielding in the tungsten targets. Tungsten self-shielding experiments were conducted in the High Flux Isotope Reactor hydraulic tube facility. The particular location in the hydraulic tube facility used for these experiments has a thermal flux of 2×10^{15} n cm^{-2} s^{-1} and an epithermal flux of 1×10^{14} n cm^{-2} s^{-1}. Results have shown that neutron self-shielding and temperature effects are responsible for the decreased yields and have led to a different target design to increase production yield.

Tungsten cross section measurements were made in the HFIR pneumatic tube facility which has a thermal flux of 4×10^{14} n cm^{-2} s^{-1} and an epithermal flux of 1.3×10^{13} n cm^{-2} s^{-1}. Calculations of ^{188}W production were based on irradiation in a HFIR PTP with a thermal flux of 1.8×10^{15} n cm^{-2} s^{-1} and an epithermal flux of 1×10^{14} n cm^{-2} s^{-1}.

Results were obtained for both ^{187}W and ^{188}W from the self-shielding experiments, but only cross sections for ^{186}W and not ^{187}W were able to be measured.

The recent HFIR measurements indicated significantly lower cross sections than reported values for the ^{187}W(n,()^{188}W reaction (the corresponding cross sections from Evaluated Nuclear Data File [NNDC 2002] are: ^{186}W: F_{th} = 37.9 b, I_0 = 485 b; ^{187}W: F_{th} = 64 b, I_0 = 2760 b), but are consistent with experience from the routine production of this medical radioisotope [Callahan et al. 1992]. Since the gamma rays of ^{188}W are exceedingly weak, ^{188}W radioactivity was determined by measuring the intensity of 155 keV gamma rays from its daughter, ^{188}Re ($t_{1/2}$ = 17.0 h), at equilibrium.

3.6 ^{166}Ho measurements

Radioactive ^{166}Ho has two isotopes: 27 h ^{166}Ho and 1200 y ^{166m}Ho. An attempt was made to measure the cross sections for capture by both of these isotopes to form radioactive ^{167}Ho. These measurements were carried out by irradiating samples of stable ^{165}Ho (100% abundance) as oxide powder and samples of radioactive ^{166m}Ho obtained from a commercial supplier.

Samples of ^{166m}Ho were irradiated in the thermal column as well as in the ICIT and CLICIT. Following the irradiations numerous gamma rays from the decay of ^{167}Ho were observed. By following the decays of these gamma rays with time, it became immediately apparent that the accepted value of the half life of ^{167}Ho, 3.1 h, was incorrect. Rather a half life of 2.88 $\pm$ 0.04 h was measured. Data on the gamma ray energies and intensities from the OSU measurements was also far superior to that available in the literature, and as a result an updated version of the decay scheme has been constructed with more precise values of the branching ratios as well as numerous new gamma rays that were not seen previously.

From the OSU measurements a thermal cross section of 3500 $\pm$ 500 b and a resonance integral of 1200 $\pm$ 200 b for ^{166}Ho^{m} was deduced. The thermal cross section disagrees with the value (9000 b) that would be deduced based on the known neutron resonances of ^{166m}Ho. One possible explanation for this discrepancy would be that the literature value for the half life of ^{166m}Ho (1200 y) is too large by a factor of 3. However, this conclusion cannot be supported from the measured data alone.

The thermal cross section and resonance integral of ^{165}Ho to form ^{166}Ho (27 h) have also been remeasured. Finally, an attempt was made to determine the cross section for capture by ^{166}Ho (27 h) to form ^{167}Ho by searching for double neutron capture by ^{165}Ho. While there is indeed evidence of ^{167}Ho in the irradiated samples of ^{165}Ho, it appears that the production of ^{167}Ho by double neutron capture can be entirely accounted for by the path that goes through ^{166m}Ho rather than ^{166}Ho.

A manuscript describing this work is in preparation for publication.

3.7 ^{47}Sc measurements

The ^{47}Ti(n,p)^{47}Sc reaction is an exothermic reaction (Q = 0.1823 MeV). A natural titanium sample was irradiated at full power (1 MW) for three hours in the CLICIT and ICIT.

Both the ICIT and CLICIT titanium irradiations yielded average fission cross sections of 15.5 ± 1.6 mb. Also, the sample was counted again after approximately nine days of decay time and gave nearly the same value (1.3 % difference) for the resultant cross section. The accepted literature value is 22.4 mb. This inconsistency warrants further investigation.

3.8 ^{91}Y measurements

The ^{91}Zr(n,p)^{91}Y reaction is an endothermic threshold reaction (Q = -0.7625 MeV) occurring in the fast flux region.

Qaim (1989) conducted a cross section measurement of all zirconium radioisotopes, including the $^{91}Zr(n,p)^{91}Y$ and the $^{91}Zr(n,p)^{91m}Y$ reactions. The present research did not measure the partial cross section to the metastable state since ^{91m}Y has a 49.7 minute half life and the sample was permitted to decay to ground state over a period of approximately 2 days prior to counting. The fission average cross section was not reported as part of Qaim's research, but was approximated from the plotted values using a normalized Watt fission spectrum as the averaging function. The approximate value is 2.4 mb over an energy range of 6.0 to 10.6 MeV.

A natural zirconium sample was irradiated at full power (1 MW) for three hours in the CLICIT and ICIT. The only expected gamma line for ^{91}Y at 1205 keV (0.3%) was not observed. Instead, the background counts were measured across the energy region where the peak would have been. The width of a nearby peak was used to approximate the peak width for determining background counts. The background counts were then used to calculate a minimum detectable activity (MDA). The MDA was used in turn to determine the largest possible peak that could go undetected. The calculated MDA was then used to calculate the largest possible cross section.

The results of this analysis were poor. The "largest possible" cross section, $< 140 \pm 26$ mb for the CLICIT irradiation and $< 106 \pm 22$ mb for the ICIT irradiation , was ~ 500 times larger than the expected value of 0.2389 mb. It is not certain why the peak was not observed or why the background was so high in that region, but it was surmised that it was related to the higher energy. Cross sections of the expected magnitude were measured for other radionuclides in this work; however, they were all low energy peaks that would more readily interact with the germanium detector.

3.9 $^{225}Ac/^{213}Bi$ measurements

Calculations were performed to investigate reactor production of ^{229}Th, the parent isotope of ^{229}Ac and ^{213}Bi, both of which are of interest in therapeutic nuclear medicine, particularly cancer therapy. A shortage of these isotopes is severely impacting this research, so increased isotope production is critical. Experimental results have shown that significant production is possible using the HFIR. Production levels surpassing the capabilities of ^{233}U processing (the current source of these isotopes) are achievable.

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